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Fatigue Properties of Borosilicate Glass Coated with Ceramics and Effect of Proof testing on Life Distribution

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Abstract

In this work, a borosilicate glass was coated with ceramic thin films by sputtering method. Roughness, hardness and static bending strength were measured as static characteristics of coated glass. Fatigue tests of coated glass were conducted under three-point bending. Although a large scatter was observed in fatigue life distributions, it was clarified that the fatigue strength was improved by coating ceramic thin films on glass. Specimens having lower lives in fatigue life distribution were adequately removed by proof testing. Therefore, it was suggested that proof testing for fatigue was effective as a screening procedure.

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*Keywords:*Fatigue; coated glass; ceramic coating film; life distribution; proof testing; bending strength; hardness; surface roughness

1. Introduction

Glass materials coated with ceramic materials by sputtering are applied to various engineering apparatuses and components. Recent works reported that glass coated with ceramic thin film is functionally used as magnetic/electronic device materials as well as optical ones. In functional and/or mechanical applications of ceramics coated glass, it is anticipated that they suffer serious damages due to cyclic mechanical or thermal stresses. Consequently, to improve the long-term durability in their practical applications, the structural design of systems using ceramics coated glass requires a fundamental understanding of their fatigue properties.

In the present work, fatigue properties of a borosilicate glass coated with alumina or silicon carbide were experimentally investigated to clarify the fatigue life properties and the effect of proof testing on life distribution. A radio frequency (RF) magnetron sputtering method was adopted in producing thin ceramic films on glass. Coated glass materials were prepared by changing a combination of RF output power and film thickness for each ceramics coated glass. Roughness and hardness of coating films, and strength of coated glass were measured as static characteristics of coated glass. Since axial loading tests, such as tensile tests, of brittle glass or ceramic materials are very difficult to be performed in evaluating their strength characteristics adequately [e.g. 1], static strength and fatigue tests were conducted under bending mode, instead of axial loading mode. The dependencies of fatigue properties of coated materials on the thickness of ceramic film and on the RF output power were also investigated to clarify a suitable sputtering condition. Finally, proof testing was carried out for coated glass materials before starting

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their fatigue tests. The effectiveness of proof testing for fatigue was discussed based on results in fatigue tests of specimens surviving after proof testing.

2. Experimental procedures

2.1. Material processing

A commercial borosilicate glass was used as a substrate material. Alumina (Al_2O_3) of 99.99% purity and silicon carbide (SiC) of 99.8% purity were adopted as target materials. The geometry of glass substrate was a disk type with a diameter of 100mm and a thickness of 2mm. In the present work, each ceramic film was coated by sputtering procedure as mentioned below, and the thickness t_f of coated film was controlled to be 1μm, 3μm or 5μm.

An RF magnetron sputtering apparatus of upper deposit type was used in the coating process. The distance between substrate and each target material was set to be 40mm in this apparatus. Substrate and target materials were being water-cooled during the processing. Before starting a steady sputtering, pre-sputtering was carried out for 300s so that a contaminated layer of target material could be removed. The initial degree of vacuum in a processing chamber was kept less than 1.3×10^{-4}Pa. The flow-rate and pressure of argon gas, which was used to activate the sputtering process in the chamber, was controlled to be 167mm^3/s and 1.3Pa, respectively. The initial temperature of substrate could be controlled, though the substrate temperature could not be controlled during sputtering of ceramic target materials. Two levels of RF output power P_{RF} were selected as P_{RF} = 400W and 600W.

Specimens of plate type with dimensions of 10mm in width and 40mm in length were cut out from coated glass and the glass substrates themselves for static strength and fatigue tests under bending mode.

2.2. Measurements of film surface characteristics

Images of surface-areas on each coating film were taken through laser scanning microscope, and the images were processed into digital data by using a personal computer in which software for image processing was installed. Data in individually measured areas were used to evaluate center-line-average roughness of their respective areas by using the images processed by the software.

The surface hardness of coated materials was measured by using dynamic microhardness tester. To avoid the influence of the substrate hardness on the film hardness, a dynamic microhardness tester is appropriate to measure the film hardness [2, 3]. The dynamic microhardness H is defined as $H = \alpha\,(F/D^2)$, in which $\alpha = 3.8584$, and F and D are respectively indentation force and depth of indenter.

2.3. Testing procedures of static bending strength

Three-point bending tests with span length of 20mm were conducted to evaluate the static bending strength of glass substrate and ceramics coated glass. The loading rate was controlled so that the rate of nominal stress at the position subjected to the maximum tensile stress in a specimen should be 100MPa/s. In setting a coated specimen on supporting equipment, the coated surface of the specimen was located in the tensile side. Fifteen specimens were prepared for each sputtering conditions. All tests were carried out in an ambient atmosphere

2.4. Procedures of fatigue tests and proof testing

Fatigue tests of glass substrate and ceramics coated glass were conducted under a sinusoidal loading at a frequency of 20Hz and a stress ratio of $R = 0.1$. The loading mode in fatigue tests and the setting specimens were as same as those mentioned in the static bending strength tests. All tests were conducted in an ambient atmosphere, and the maximum stress σ_{max} generated in each specimen was controlled to be 70MPa or 80MPa during the fatigue tests. Fifteen specimens were tested in each test condition of respective material. Each fatigue test was stopped if no failure occurred after 1.728×10^6 cycles (24 hours).

Proof testing was conducted using a triangular wave with constant stress-rates of 100MPa/s in loading and −100MPa/s in unloading. The proof stress σ_p was set to be 100MPa. Subsequent fatigue tests of specimens surviving after proof testing were conducted under the same conditions as the aforementioned fatigue test.

3. Experimental results and discussions

3.1. Characteristics of coating films

The comparison with the mean surface roughness of glass substrate clarified that the surface coated with ceramics in every case was rougher than the glass surface. The roughness of coated film was found to increase with increasing the film thickness t_f. It is well recognized that such a tendency is caused by a shadowing effect occurring in a coating process of physically vaporized deposit type. As a whole trend in Al_2O_3 and SiC coated materials, the surface of film coated under lower RF output power P_{RF} became smoother than the surface of film coated under higher power.

Every coated film was found to be harder than the glass substrate. In each ceramic film, thicker film had higher hardness, because the effect of softer glass substrate on hardness of coated film decreased in thicker film. It was also clarified that the surface hardness of films coated under a higher RF output power P_{RF} was higher. Consequently, it is suggested that higher RF output power brings denser film.

3.2. Strength properties of coated materials

Table 1 summarizes average values of bending strength σ_f of coated glass. Since the mean strength of glass substrate itself is 88.5MPa, it is concluded that the strength of glass is improved by ceramic coating. As seen in Table 1, the strength of coated glass increases as the film thickness becomes thicker. This trend is more clearly seen in SiC coated glass.

Table 1. Bending strength of coated glass in MPa

(a) Al_2O_3 film	Film thickness (t_f)		
RF output power (P_{RF})	1μm	3μm	5μm
400W	95.9	101	101
600W	97.2	99.4	99.5

(b) SiC film	Film thickness (t_f)		
RF output power (P_{RF})	1μm	3μm	5μm
400W	90.6	94.5	98.6
600W	91.9	96.6	98.6

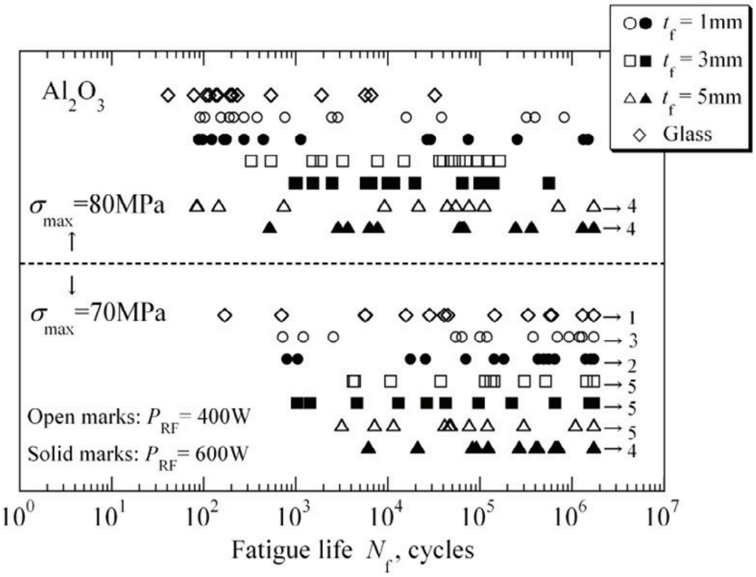

Fig. 1. Fatigue life correlated with applied stress in glass substrate and Al_2O_3 coated glass

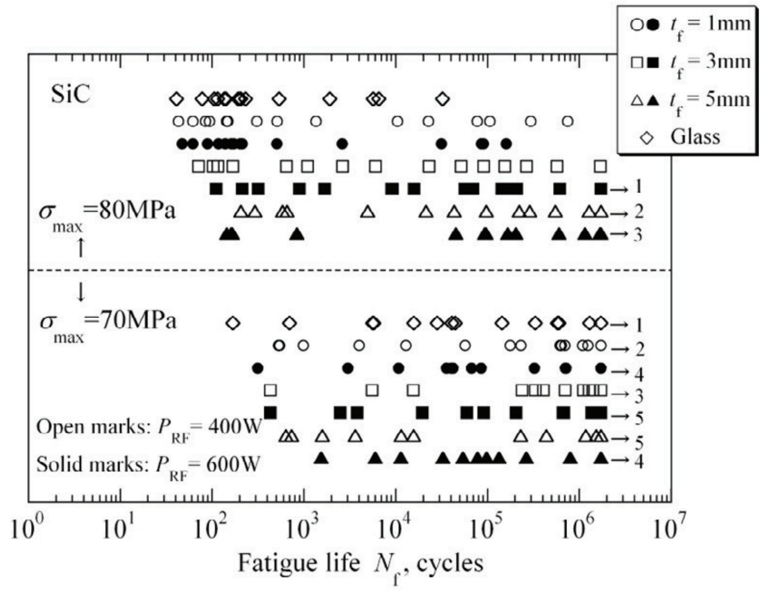

Fig. 2. Fatigue life correlated with applied stress in glass substrate and SiC coated glass

3.3. Fatigue properties of coated glass

Fatigue lives N_f of glass substrate as well as Al_2O_3 and SiC coated glass under two stress levels are shown in Figs. 1 and 2. The number attached to the arrow in the figures presents the number of specimens surviving at the truncated number of cycles, i.e., 1.728×10^6. As expected obviously, the number of surviving specimens increases under lower applied stress. As seen in the figures, the fatigue life has also a large scatter in its distribution, similarly to a scatter observed in the static strength of ceramic materials. It is revealed from these figures that the minimum fatigue life in every coated glass is longer than that in glass substrate and glass coated with thicker film has longer life.

Examples of statistical life distribution of specimens broken in fatigue test, which is plotted on Weibull probability paper, are shown in Figs. 3 and 4. Curves in the figures show life distributions $F(N_f)$ obtained by fitting them to three-parameter Weibull distribution function, i.e., $F(N_f) = 1 - \exp\{-[(N_f - N_L)/N_S]^m\}$. Three-parameter Weibull distribution function consists of scale parameter N_S, location parameter N_L , and shape parameter m.

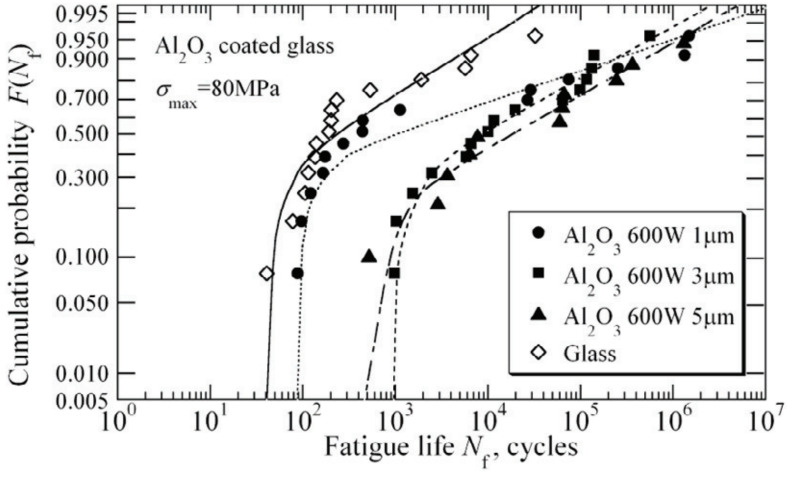

Fig. 3. Examples of life distributions of broken specimens plotted on Weibull probability paper in Al_2O_3 coated glass

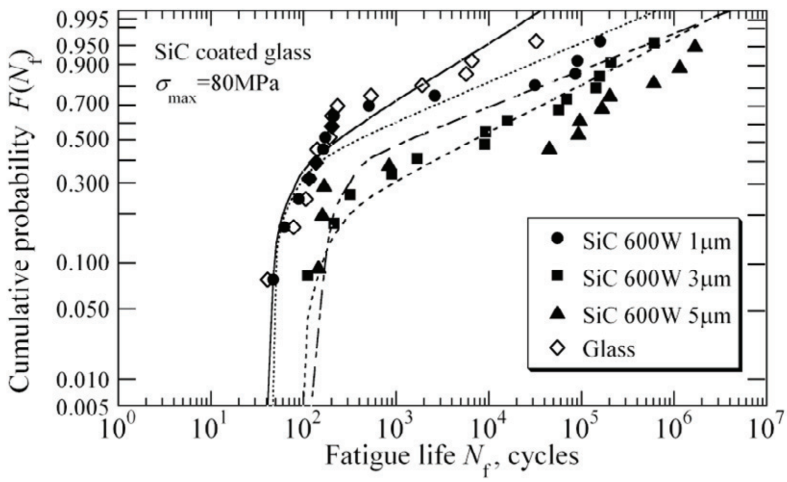

Fig. 4. Examples of life distributions of broken specimens plotted on Weibull probability paper in SiC coated glass

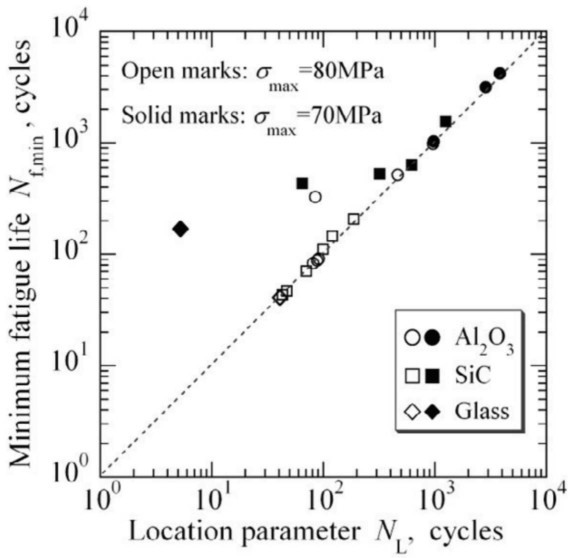

Fig. 5. Minimum fatigue life correlated with location parameter in fitted function of three-parameter Weibull distribution

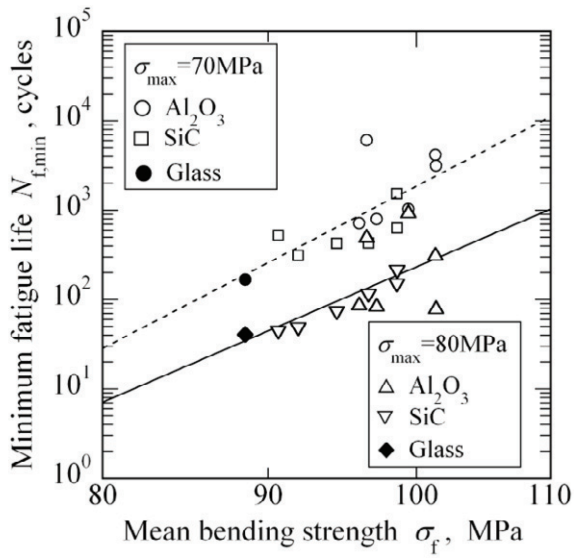

Fig. 6. Minimum fatigue life correlated with mean values of static bending strength

The location parameter N_L implies the minimum life expected in the fitted life distribution under consideration. Figure 5 presents the relation between location parameter and observed minimum fatigue life $N_{f,min}$ in every material. A good coincidence between them is seen in Fig. 5. However, location parameters in a few data shift toward a lower life side. In these situations, life distributions in experiments are not adequately fitted to three-parameter Weibull distribution function. On the other hand, in Fig. 6, $N_{f,min}$ is also correlated with mean bending strength σ_f. The straight line in the figure shows the relation of $N_{f,min}$ approximated as a power function of σ_f. Although some dispersion is seen, the variation of $N_{f,min}$ depending on σ_f is appropriately expressed by the relation.

4. Effect of proof testing on life distribution

Life distributions after proof testing are depicted in Fig. 7, in which cross marks present the minimum lives of respective materials in ordinary fatigue tests. In each material, the minimum life after proof testing is longer than the life corresponding to the cross mark. Consequently, it may be concluded that the proof testing conducted under the condition set in this work is effective in removing specimens having lower fatigue lives. This coincides with the result in bulk ceramic materials that the proof testing is effective for screening weaker specimens under fatigue [4].

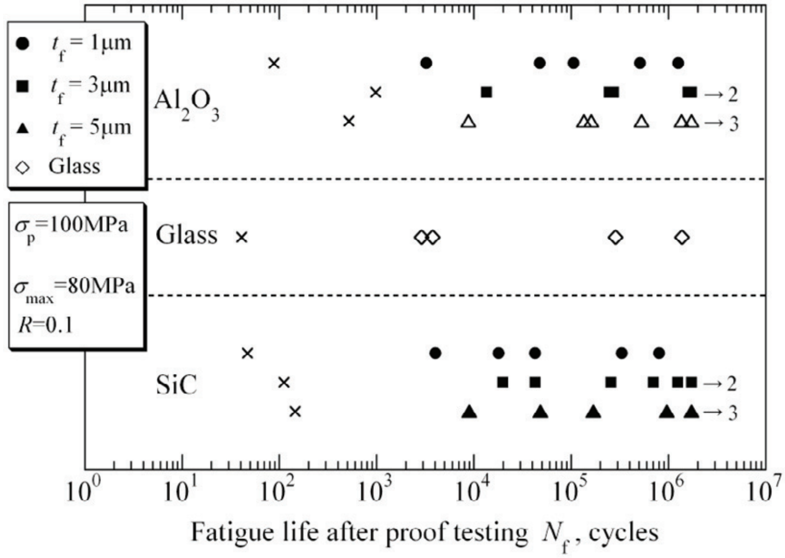

Fig. 7. Life distribution after proof testing

5. Conclusions

The long-term durability of coated materials with high performance should be adequately evaluated prior to their application. This work aims at clarifying fatigue property as one of the durability characteristics of such materials. In the present work, a borosilicate glass was coated with alumina or silicon carbide thin films by a radio frequency (RF) magnetron sputtering method, and fatigue lives of ceramics coated glass were investigated. Under RF output power of 400W or 600W, the thickness t_f of coated film was prepared to be 1μm, 3μm or 5μm. Roughness and hardness were measured as surface characteristics of ceramic films coated on glass substrate. As a whole trend, the surface of film coated under 400W was smoother than the surface of film coated under 600W. The surface hardness of films coated under 600W was higher than that in coating under 400W. The strength of coated glass was evaluated under three-point bending mode. It was found that the strength of glass was improved by coating ceramic thin films on glass. Fatigue tests of coated glass were conducted under three-point bending mode and at stress ratio of 0.1. Although a large scatter was observed in experimental life distributions, it was clarified that the fatigue strength was improved by coating ceramic thin films on glass. Fatigue life distribution in glass coated with thicker film was found to shift toward longer life region. Minimum fatigue life of each coated glass was well expressed as a power function of the mean strength. Lower life region in fatigue life distribution was adequately truncated by proof testing. It was concluded that the proof testing was an effective procedure in screening out fatigue lives of ceramics coated glass.

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